CCQM Activities in the Inorganic and Electrochemical Analysis Working Groups

Since 1997, the NIST's Analytical Chemistry Division has participated in 34 of 53 studies of the Consultative Committee for Amount of Substance (CCQM) organized by the Inorganic Analysis Working Group (IAWG) and 11 of 15 studies organized by the Electrochemical Analysis Working Group (EAWG). The CCQM conducts international comparisons to establish equivalence among measurements made by national metrology institutes (NMIs). Studies conducted during the past year included measurement of a wide variety of elements in materials related to the *environment, food and nutrition, metals and purity analysis, geological and advanced materials, and pH..*

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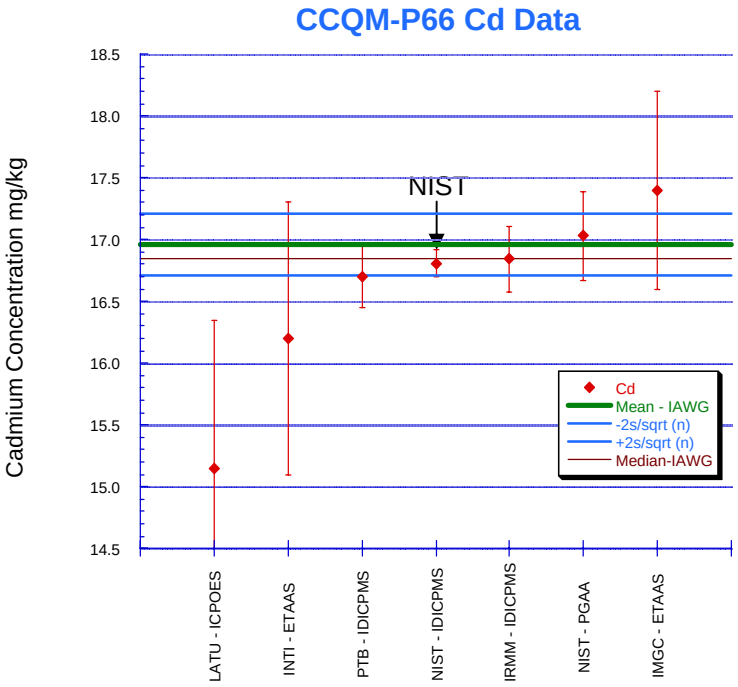
During the past few years, NIST participated in CCQM studies in areas related to the environment, food and nutrition, metals and purity analysis, as well as geological and advanced materials. NIST coordinated the following CCQM studies within the IAWG during 2006: CCQM-P66 Metals in Fertilizer and CCQM-K49/P85 Essential and Toxic Elements in Bovine Liver.

Pilot Study CCQM-P66 was the first study of a fertilizer-based material, and proved to be quite challenging. Seven NMIs and one expert laboratory participated in this study. The data for Cd, shown in the figure, demonstrated the best agreement for any of the elements studied. Only NIST and the Institute for Reference Materials and Measurements (IRMM) provided data for all five elements in CCQM-P66. It is not clear at this time whether or not there will be a follow-up key comparison.

Key comparison CCQM-K49 and an associated pilot study, CCQM-P85, are currently in progress, with a deadline for data reporting in February 2007. The studies include seven toxic and essential elements that present varying degrees of difficulty in their determination, from relatively easy (Fe and Zn), to moderately difficult (Se, Pb and Cd) to very difficult (As and Cr). In addition, CCQM-K49/P85 represents a first for the IAWG in that all NMIs with inorganic analysis capabilities were requested to participate in either the key comparison or the pilot study for at least two designated elements, iron and zinc. In view of the large number of studies undertaken each year, participation by NMIs in any one particular study has dramatically decreased, limiting the ability to compare measurement capabilities. It has been decided that one study per year will be designated as an exemplary study, in an attempt to maximize participation in a common study to enable a direct comparison of NMI capabilities.



Ten NMIs and 5 expert laboratories participated in CCQM-P86 Total Se and Se Speciation Analysis of Pharmaceutical Supplements. NIST submitted results for both total Se and selenomethionine, which appeared to be in good agreement with other results submitted (no formal report has been made available to date). A Key Comparison is planned to follow up on this study, but at lower Se levels. Wheat flour containing a total Se content of about 1 mg/kg (total Se) was proposed.

In addition, we recently applied a double spike technique developed here at NIST (Mann and Kelly, 2005) in a CCQM pilot study (CCQM P75) to determine the $\delta^{34}S$ of the essential amino acid methionine. We had previously improved a specialized high-accuracy mass spectrometric method to precisely measure the isotopic composition and concentration of sulfur in small samples (<1 μmole S). The uncertainty for our results in the CCQM study (reported as combined uncertainties) was smaller then that reported by the five other laboratories that participated and

on half the amount of sample (100 μg). Additionally, two other measurements in which we used a factor of 10 less sample (10 μg) also fall within in our uncertainty reported, again showing the capability of the technique for measuring small sample sizes.

NIST participated in several additional pilot studies and key comparisons with good results including CCQM-K42 Constituents of an Aluminum Alloy, CCQM-P64 Trace Elements in Soybean Powder, and CCQM-P62 Purity Analysis of Nickel Based on 6 Metallic Elements.

Under the auspices of the Electrochemical Analysis Working Group, NIST participated in three Key Comparisions: two in the area of pH and one in the area of electrolytic conductivity: CCQM-K18 Carbonate Buffer, pH 10; CCQM-K19 Borate Buffer, pH 9.2; and CCQM-K36a/K36b Electrolytic Conductivity, 0.5 S/m and 5 mS/m. In addition, results for CCQM-K34 Potassium Acid Phthalate, Amount Content of Weak Acid, conducted in 2004, were approved by the BIPM for the Key Comparison Database.

Impact: The results from these and future studies will be used to benchmark the appropriate Comparability and Measurement Claims (CMCs) of the participating NMIs.

Future Plans: NIST plans to participate in the following IAWG and EAWG comparisons in FY07: CCQM-K49 Toxic and Essential Elements in Bovine Liver; CCQM-K56 Elements in Soybean Powder; CCQM-K57 Elements in Clay; CCQM-K58 Nitrogen and Trace Elements in Silicon Nitride; CCQM-K43.1 (Subsequent) Methylmercury in Swordfish;, CCQM-P97 Cadmium and Lead in Herbs; CCQM-K59 Nitrate Calibration Solution; CCQM-K48 Potassium Chloride; CCQM-K20 Tetroxalate Buffer, pH 2, organized by NIST; and CCQM-P83 Electrolytic Conductivity, 0.5 mS/m.

Reference: Mann, J. L. and Kelly, W. R. (2005) ***Measurement of sulfur isotope composition ($\delta^{34}S$) by multiple-collector thermal ionization mass spectrometry using a ^{33}S-^{36}S double spike.*** *Rapid Commun. Mass Spectrom.*, **19**: 3429–3441.